H. PRICE

varian 2200/2300 series spectrophotometers

DIFFUSE REFLECTANCE ACCESSORY WITH DETECTORS

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varian

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DIFFUSE REFLECTANCE ACCESSORY WITH DETECTORS

1.0 GENERAL INFORMATION

1.1 INTRODUCTION

The In-Cell Space Diffuse Reflectance accessory is used to obtain spectra of a scattering material, liquid or solid, over wide solid angles in either a reflecting or transmitting mode. Absorption spectra of turbid, translucent, or opaque refractory materials may be measured where standard techniques prove inadequate due to loss of light or inclusion of scatter components unrelated to the absorption of the substance.

1.2 DESCRIPTION

A beam of light from the monochromator is alternately directed down the sample and reference paths (see Figure 1). The sample beam (at the rear of the instrument) is incident on M1, which reduces the size of the beam and directs it to the plane mirror M2. M2 directs the beam towards the integrating sphere entrance port, P1, at an angle of 3° 20'. Lens L1 further keeps the beam size within bounds. L1 is rotated slightly to keep the specularly reflected beam from the sample from reentering the sphere after reflection from the front and rear surfaces of the lens. This reflection only exists when the attitude of the sample is such as to exclude the specular component from the integrating sphere.

The sphere cap (Figure 2) has two positioning marks: D and S. When the sphere cap is oriented so that the D is facing up, the specular component of the radiation reflected from the sample is excluded from the light reaching the detector; the system then measures only the diffuse reflectance. When the sphere cap is oriented so that the S is facing up, the specular component is included in the measurement.

When the specular component is excluded (D position), the beam strikes the sample surface normally. When the specular component is included (S position), the angle of incidence is 3° 20'.

The reference beam (at the front of the instrument) enters the integrating sphere at P3 and strikes a small target coated with barium sulfate ($BaSO_4$) paint. The target then diffusely illuminates the sphere during the reference time interval.

The photomultiplier and lead sulfide detectors mounted on top of the sphere view the interior of the sphere, which is alternately illuminated by sample and reference light.

Two methods for clamping the sample into place are provided when making reflectance measurements. The large clamp is convenient when large heavy samples require considerable pressure to keep the sample in position at the sample port P2. A more lightly loaded clamp for small delicate samples is attached to the sphere cap so that the sample position will not be changed when rotating the sphere cap through 180°.

Samples of diffusely transmitting material may be mounted in the 3-inch space between the lens and the entrance port P1. Care should be taken to avoid contamination of the lens by the sample or by the operator.

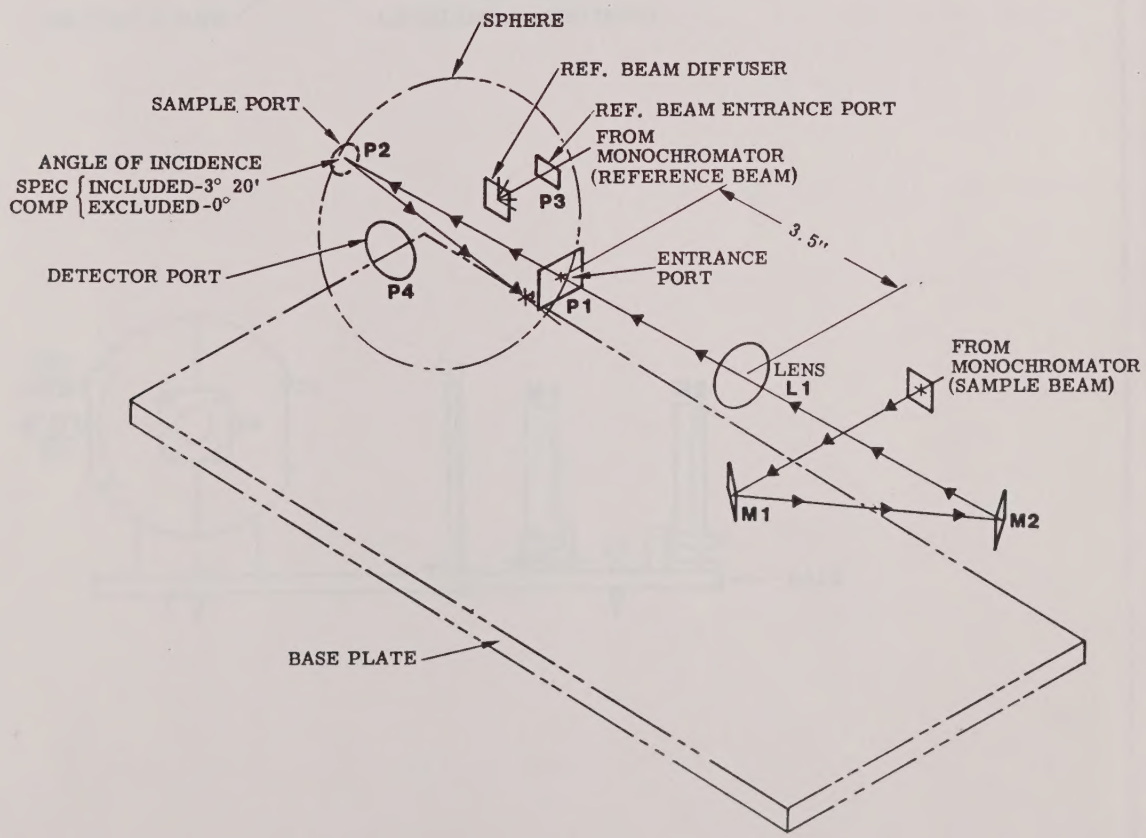


Figure 1. Diffuse Reflectance Accessory, Isometric Optical Layout

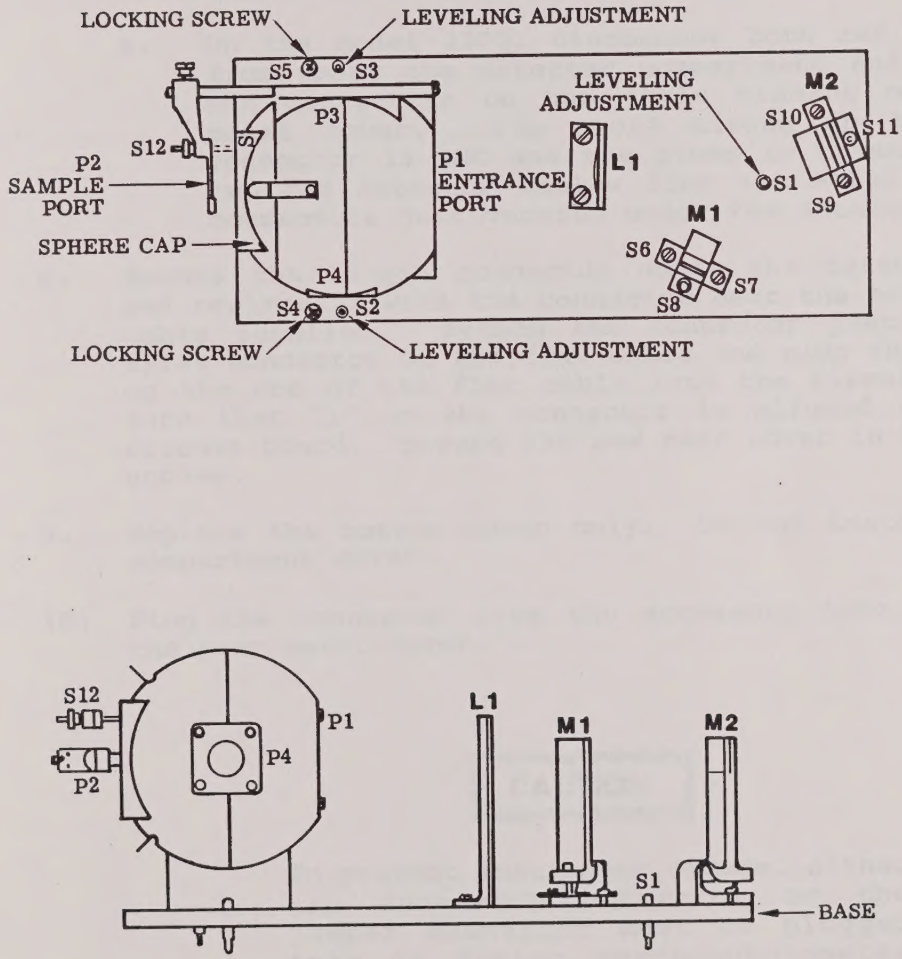


Figure 2. Diffuse Reflectance Accessory Top and Side Views

7. Depending upon the model (2200 or 2300), proceed as follows:

   a. On the Model 2200, disconnect the red dynode cable from under the detector compartment and plug it into the connector on the angle bracket of the new rear metal cover. Plug the red dynode cable from the metal cover into the connector just vacated under the detector compartment.

   b. On the Model 2300, disconnect both red detector cables from under the detector compartment and plug them into the connectors on the angle bracket of the new rear metal cover. (To avoid mixing up the cables, one connector is BNC and the other is threaded.) Plug the two red detector cables from the metal cover into the connectors just vacated under the detector compartment.

8. Remove the 15-pin connector under the detector compartment and replace it with the connector near the center of the flat cable supplied. Attach the connector just removed to the first connector on the flat cable and plug the edge connector on the end of the flat cable into the circuit board, making sure that "1" on the connector is aligned with "1" on the circuit board. Secure the new rear cover in place with three screws.

9. Replace the bottom cover only. Do not install the detector compartment cover.

10. Plug the connector from the accessory into connector J4 on the rear metal cover.

CAUTION

To prevent instrument damage, either the accessory connector or the jumper connector must be plugged into J4 during spectrophotometer operation.

1.5 MECHANICAL ALIGNMENT PROCEDURES

1. Turn the ACCESSORY switch on the rear panel to ON and power up the spectrophotometer. Set the LAMP SELECT parameter to VIS, set the REFERENCE MODE to AUTO GAIN, and set the slit (SBW) to 3.6 nm. Open the lamp access door (above the sample compartment) and turn the BEAM HEIGHT knob to 1/3. Press GO

TO WAVELENGTH and enter 185 nm. When the instrument is at 185 nm, press GO TO WAVELENGTH again and enter 0 nm.

2. Observe the beam on the mirrors. If the beam is not clipping and near center on the reference port P3, centered on mirrors M1 and M2, and if the beam is near center on entrance port P1 and sample port P2, no further adjustment is necessary. Using the spring loaded screws (see Figure 2), lock the accessory baseplate into position and proceed to Section 1.6, High Absorbance Adjustment. Otherwise, continue to the next step.

3. If the beam is not centered on reference port P3, adjust the level-adjusting screws S2 and S3 until the beam is near center and not clipping the sides of the port. After adjusting S2 or S3, measure the distance from the top of the accessory mounting plate to the compartment base near the respective screws to be sure the plate is level.

4. If the beam falls off the surface of mirror M1, adjust leveling screw S1 to center the beam vertically. Loosen the two binder-head screws S6 and S7 holding the horizontal retaining bar at the base of M1. Move M1 until the beam is as near the center of M1 as possible and tighten the bar until some holding friction is exerted on the mount, yet the mount can still be rotated.

5. To adjust the angle of M1, first look through the sample port P2, centering the entrance port P1 in your field of view. Rotate M1 until the beam strikes M2 so that the beam is aligned horizontally with respect to the center of M2 (see Figure 3). Tighten the horizontal bar with the two binder-head screws. Adjust the Allen-head set screw S8 at the rear of the base of the M1 mount until the beam on M2 is centered vertically. (NOTE: The position of lens L1 is set at the factory and should need no further adjustment.)

6. To adjust the angle of M2, place the mylar target at the sample port P2. Loosen the two binder-head screws S9 and S10 so that M2 is free to rotate. Twist M2 until the beam observed at P2 is centered horizontally and tighten S9 and S10. Adjust the Allen-head screw S11 until the beam at P2 is centered vertically.

7. Return to step 2.

TEST FUNCTION
ENTER 30349
SELECT FILTER #1
V VIS DET
VIS GAIN

1.6 HIGH ABSORBANCE ADJUSTMENT

WARNING

The high-absorbance adjustment requires exposing a printed circuit board containing high voltage. Only qualified service personnel should make the adjustment.

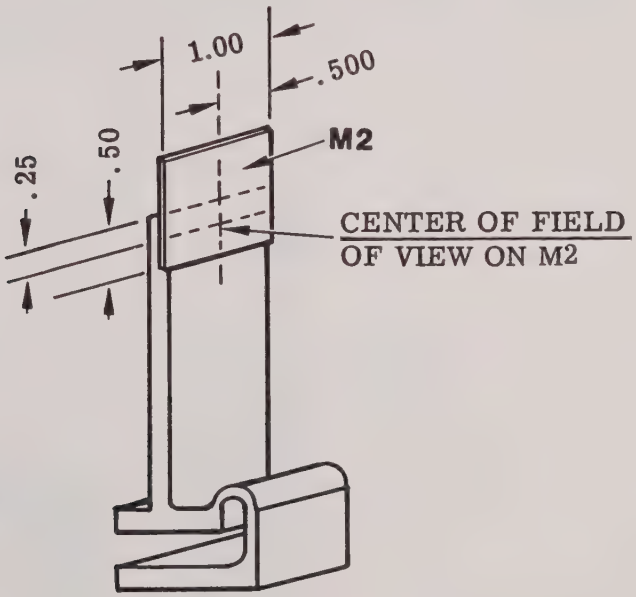


Figure 3. Field of View on Mirror M2

1. Disconnect the spectrophotometer power line from the line power outlet. Unscrew and remove the cover over the ramp board on the right-hand side of the detector compartment. Check that the vertical board that is exposed has "RAMP BOARD" printed on it. Attach an oscilloscope with a minimum of 10-megohm probe impedance to TP9 and TP34 (ground). Set the vertical sensitivity to 0.01 volt/cm and the sweep to 10 msec/cm.

2. With the accessory in place, power up the spectrophotometer. Check that the ACCESSORY knob on the rear cover is switched to ON. Open the lamp access door and set the BEAM HEIGHT to

1/3. Press GO TO WAVELENGTH and enter 500. Set the other instrument parameters as follows:

ORDINATE	ABSORBANCE
ABSCISSA	WAVELENGTH
REFERENCE MODE	AUTO GAIN
SBW (SPECTRAL BANDWIDTH)	3.0 NM
BEAM INTERCHANGE	REVERSE
LAMP POWER	BOTH ON
LAMP SELECT	AUTO
DETECTOR SELECT	AUTO

3. Remove the front sample compartment cover. Locate R14, the UV/VIS adjusted potentiometer, along the edge of the accessory printed circuit board on the right of the sphere (see Figure 4). Block the rear beam and drape the front of the sample compartment with a black cloth to prevent light from entering the compartment. With a small screwdriver, adjust R14 to remove the pedestal from the waveform. If the pedestal cannot be removed, move J1 (directly below R14) to the unused pin and adjust R14 again.

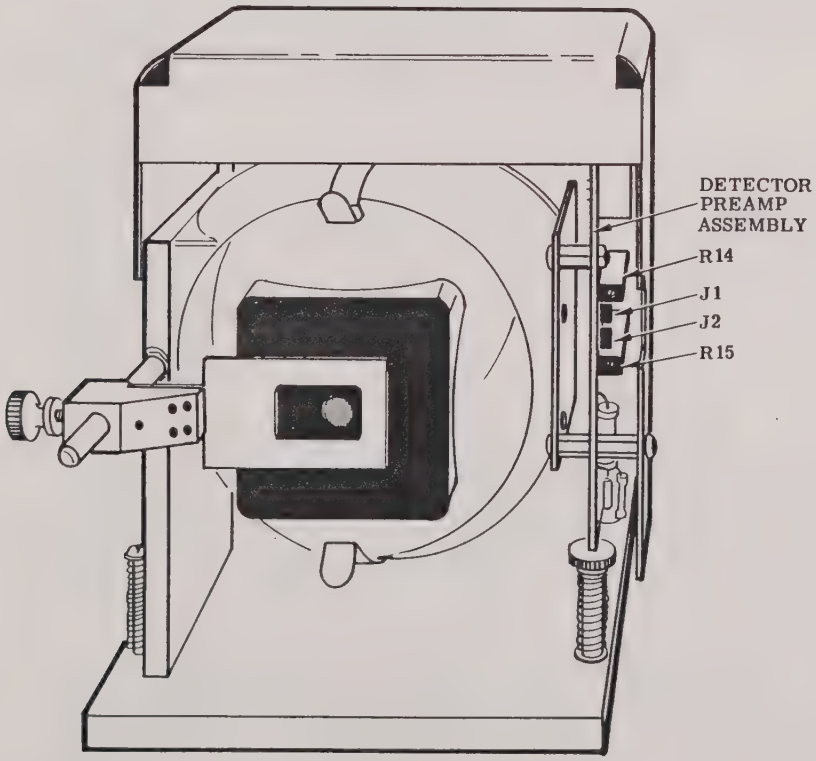


Figure 4. Location of UV/VIS and NIR Adjustments

4. On the Model 2300, press GO TO WAVELENGTH and enter 1600 nm. Set GAIN so the SBW is approximately 13 nm. Set the oscilloscope vertical sensitivity to 0.1 volt/cm. Locate R15, the NIR potentiometer (see Figure 4). In a similar fashion to Step 3, adjust R15 to remove the pedestal. If the pedestal cannot be removed, move J2 (directly above R15) to the unused pin and adjust R15 again.

5. Disconnect the spectrophotometer from the line power. Unblock the rear beam. Remove the oscilloscope probes and replace the ramp board cover. Remove the three screws securing the rear panel. Replace the detector compartment cover and secure it in position with one screw at the rear of the detector compartment, then replace the rear panel. Replace the front sample compartment cover.

1.7 TESTING FOR THE SPECULAR COMPONENT

1. Repower the spectrophotometer. Open the lamp access door and turn the BEAM HEIGHT knob to the 1/3 position. Press GO TO WAVELENGTH and enter 546 nm. Set the other instrument parameters as follows:

ORDINATE	TRANSMISSION
ABSCISSA	WAVELENGTH
REFERENCE MODE	AUTO GAIN
SBW (SPECTRAL BANDWIDTH)	3.6 NM
PEN RANGE	0/100
RESPONSE TIME	1.0 SEC
BEAM INTERCHANGE	REVERSE
LAMP POWER	BOTH
LAMP SELECT	AUTO

2. Position the sphere cap so that the screw of the clamp assembly S12 on the spere cap is in the upper right-hand corner with the D mark facing up. Place a sample of $BaSO_4$ or equivalent reflecting material over the sample port P2. (NOTE: To avoid scratching delicate mirrors, use a single layer of scotch tape around the sample port P2.)

3. Replace the covers to the sample compartment. Press the AUTOBALANCE key so that the display reads 100% T. Place a good quality mirror over P2. Close the covers again. The reading should not exceed 1.5% T. If the reading is greater, either the specularly reflected beam is not exiting the sphere at P1, dirt on the surface of lens L1 is scattering the specularly reflected beam from the sample mirror back into the sphere, or the sample mirror does not have a good specular surface. Check the location of the beams at P1 and P2 and readjust M2 if necessary to center the beams in P1 and

P2, after removing the sample cover.

Rotate the sphere cap 180° so that the S mark is facing up. This is done by rotating the lower spring clamp to disengage it from the slot in the sphere cap and then lifting the upper clamp out of the upper slot while removing the sphere cap. Replace the covers. The display should read approximately 90% T.

1.8 CHECK FOR OPERABILITY

1. Change the ORDINATE to ABSORBANCE, PEN RANGE to 1.0; otherwise leave the same instrument parameter settings as Step 1 in Section 1.7 above. Rotate the sphere cap so the D mark is facing up.

2. Place a reflectance standard at the sample port P2.

3. Close the sample compartment covers. Set the ZERO SUPPRESSION value (part of the PEN LIMITS parameter) to -0.5 ABS. Press AUTOBALANCE. Set the SCAN RATE to 2 nm/sec and CHART DISPLAY to 20 nm/cm.

4. Depending upon the model (2200 or 2300), proceed as follows:

   a. On the Model 2200, scan to 880 nm and record a baseline correction from 880 to 220 nm.

   b. On the Model 2300, change the REFERENCE MODE to AUTO SELECT, then scan to 801 nm. Set GAIN to give a SBW of approximately 13.5. Record a baseline correction form 1800 to 220 nm.

5. Set the ORDINATE to % TRANSMISSION and record the corrected baseline.

6. If excessive baseline discontinuity appears at the filter change points, check for beam clipping at the reference beam entrance port P3 and if necessary, readjust the S3 leveling screw. Recheck the test for specular component after adjustments.

2.0 OPERATION

NOTE

At all times, the operator must take care to avoid contamination of the interior of the sphere, the mirrors, and the lens.

1. Check the rear panel for the following:

   a. The ACCESSORY switch is in the ON position.

   b. The accessory cable, not the jumper, is in connector J4.

2. Select a reflectance standard of high-quality, stable $BaSO_4$ paints or powders. Be sure the selected standard will not contaminate the integrating sphere in any way.

3. With the reflectance standard in the sample port P2 and the compartment covers closed, run a baseline with the ORDINATE set to ABSORBANCE as described in the operator's manual for the spectrophotometer. Then check the baseline with the ORDINATE set to % TRANSMISSION.

4. To measure an actual sample, set the ORDINATE to % TRANSMISSION and the other instrument settings parameters as desired. Position the sphere cap to either the D mark (diffuse, specular component excluded) or the S mark (specular component included).

5. Position the reference sample in the clamp desired, close the covers, and press the AUTO BALANCE key. The monitor should display 100% T.

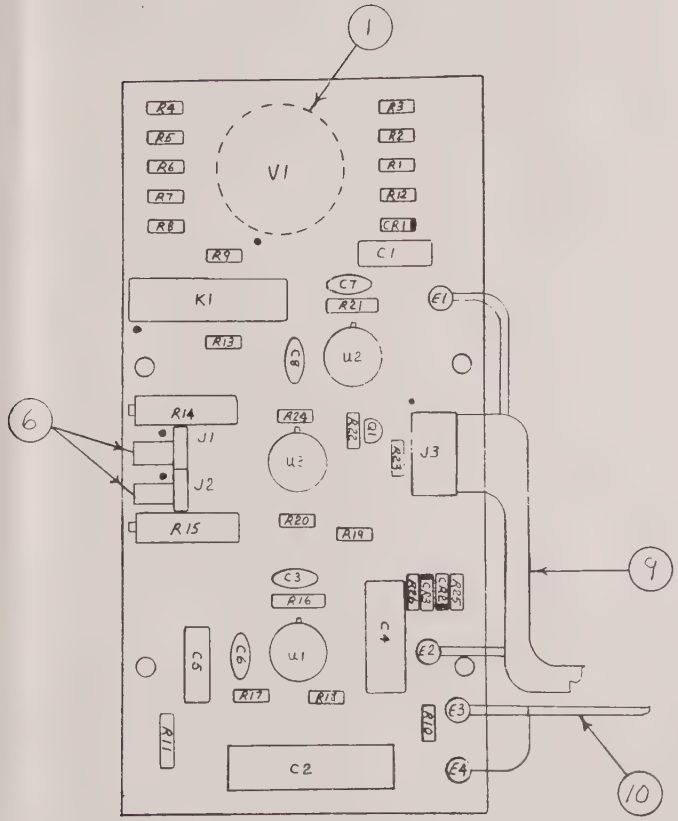
6. Remove the reference sample and replace it with the sample to be measured. Close the covers. The measurement will be displayed directly in %T.

7. A sample of diffusely transmitting material can be mounted in the 3-inch space between the lens and the entrance port P1.

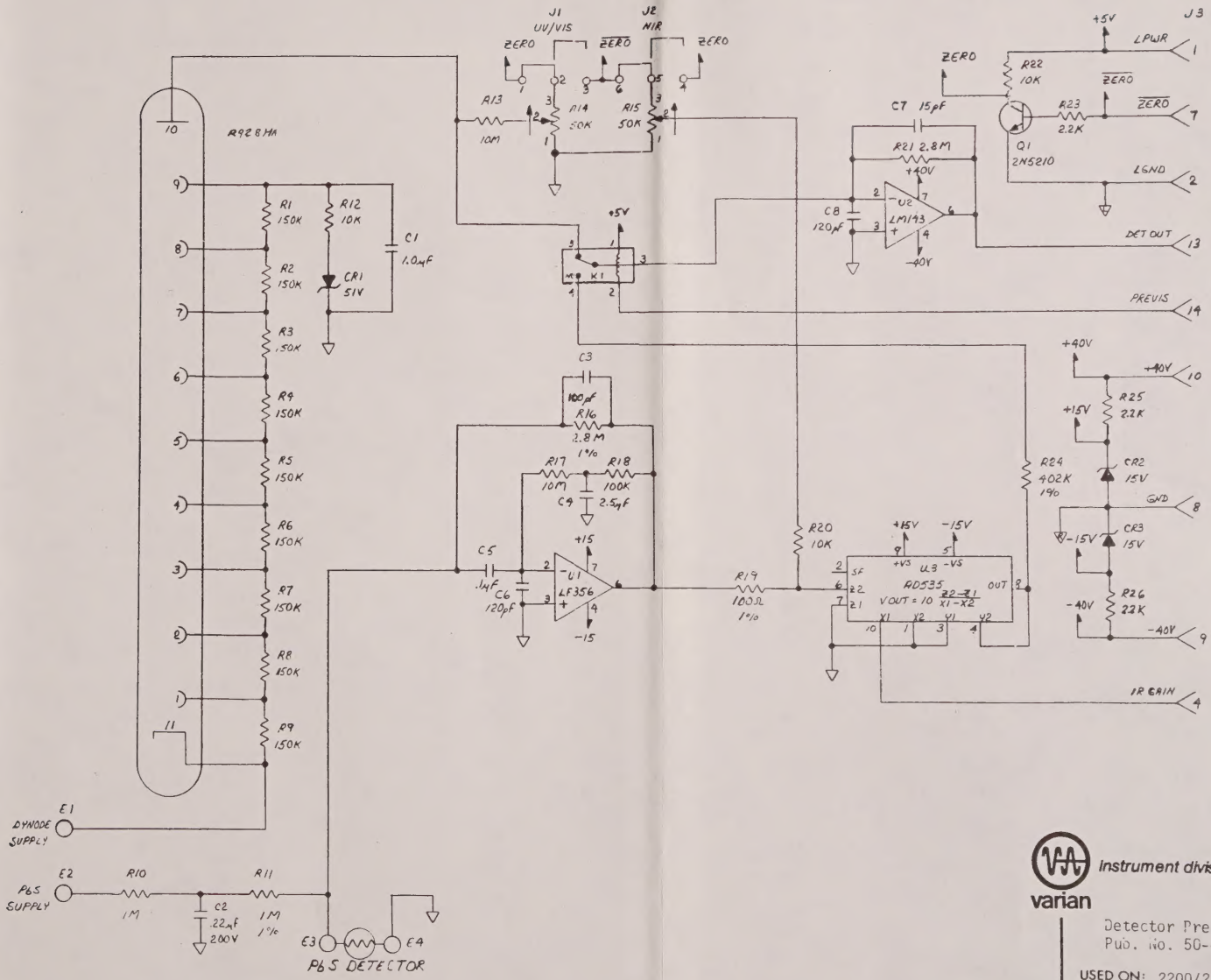
8. To remove the accessory,

   a. Turn the spectrophotometer power off.

   b. Disconnect the accessory cable from connector J4 on the rear panel.



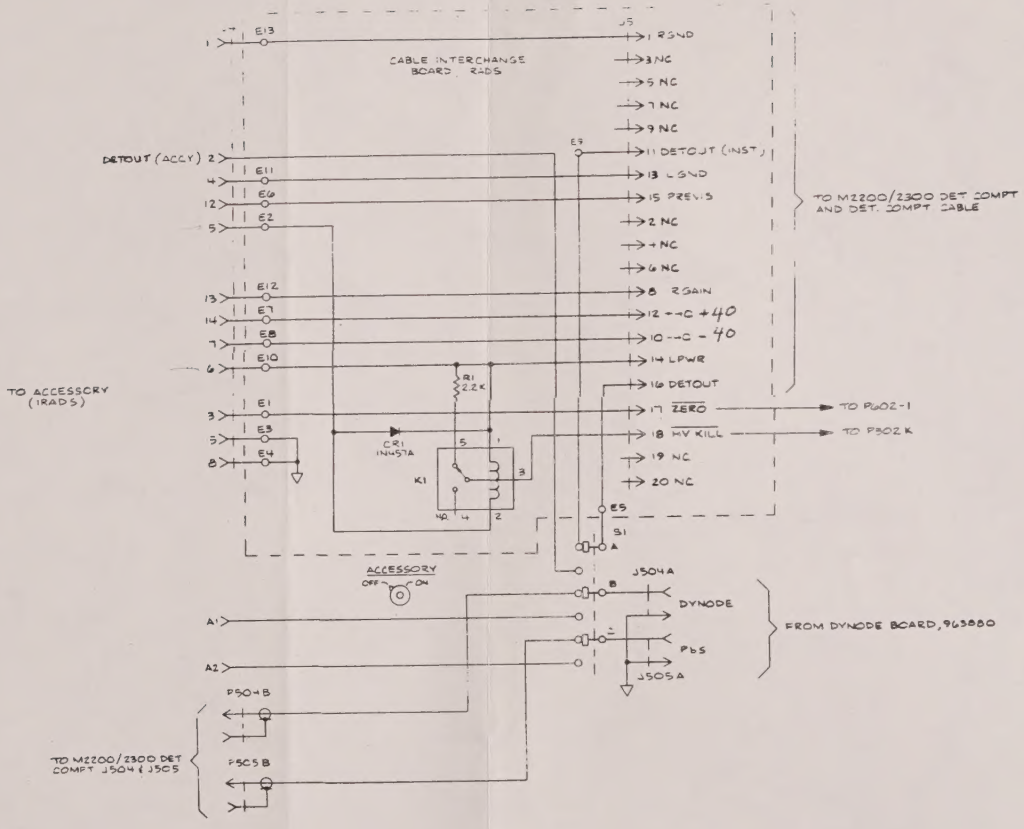
SCHEM REF	VARIAN PART NO	DESCRIPTION
1	B952881	Socket,Tube,11-pin
2	B952891	Tube,Photomultiplier,Hamamatsu R928HA
3	50-413211	PCB,Detector Preamp
4	50-413212	Schematic,Detector Preamp
5	51-910065	Terminal,PC
6	51-452933	Connector,Shorting,2-pin
7	62-899954	Pad,IC,8-pin
8	62-899956	Pad,IC,10-pin
9	50-413213	Cable,Sample Compartment
10	50-413215	Cable,PbS
C1	41-718805	Cap,Mylar,1.0uF,100V
C2	41-726994	Cap,Mylar,0.22uF,200V
C3	41-159586	Cap,Mica,100pF,500V
C4	41-718801	Cap,Mylar,2.5uF,100V
C5	41-724964	Cap,Mylar,0.1uF,200V
C6	41-159565	Cap,Mica,120pF,500V
C7	41-159506	Cap,Mica,15pF,500V
C8	41-159565	Cap,Mica,120pF,500V
CR1	66-314757	Diode,Zener,51V,1N4757A
CR2	66-314744	Diode,Zener,15V,1N4744A
CR3	66-314744	Diode,Zener,15V,1N4744A
J1	51-407210	Connector,Strip,Right Angle
J2	51-407210	Connector,Strip,Right Angle
J3	51-108876	Connector,DIP,14-socket
K1	72-569947	Relay,Reed,1-form C
R1	32-301615	Resistor,Fxd,CC,150k,5%,1/4W
R2	32-301615	Resistor,Fxd,CC,150k,5%,1/4W
R3	32-301615	Resistor,Fxd,CC,150k,5%,1/4W
R4	32-301615	Resistor,Fxd,CC,150k,5%,1/4W
R5	32-301615	Resistor,Fxd,CC,150k,5%,1/4W
R6	32-301615	Resistor,Fxd,CC,150k,5%,1/4W
R7	32-301615	Resistor,Fxd,CC,150k,5%,1/4W
R8	32-301615	Resistor,Fxd,CC,150k,5%,1/4W
R9	32-301615	Resistor,Fxd,CC,150k,5%,1/4W
R10	32-301710	Resistor,Fxd,CC,1M,5%,1/4W
R11	31-227100	Resistor,Fxd,MF,1M,1%,1/4W
R12	32-301510	Resistor,Fxd,CC,10k,5%,1/4W
R13	32-301801	Resistor,Fxd,CC,10M,5%,1/4W
R14	36-792210	Resistor,Var,Cermet,50k,15T
R15	36-792210	Resistor,Var,Cermet,50k,15T
R16	31-607280	Resistor,Fxd,MF,2.8M,1%,1/4W
R17	32-301801	Resistor,Fxd,CC,10M,5%,1/4W
R18	32-301610	Resistor,Fxd,CC,100k,5%,1/4W
R19	31-223100	Resistor,Fxd,MF,100 ohm,1%,1/4W
R20	32-301510	Resistor,Fxd,CC,10k,5%,1/4W
R21	31-607280	Resistor,Fxd,MF,2.8M,1%,1/4W
R22	32-301510	Resistor,Fxd,CC,10k,5%,1/4W
R23	32-301422	Resistor,Fxd,CC,2.2k,5%,1/4W
R24	31-226402	Resistor,Fxd,MF,402k,1%,1/4W
R25	32-301422	Resistor,Fxd,CC,2.2k,5%,1/4W
R26	32-301422	Resistor,Fxd,CC,2.2k,5%,1/4W
U1	62-600688	IC,Op Amp,LF356H
U2	62-601526	IC,Variable Amp,AD535JH
U3	62-601665	IC,Op Amp,LM143H



varian instrument division

Detector Preamp
Pub. No. 50-413-212

USED ON: 2200/2300 Accessory
ASSY NO: 50-413-210



SCHEM REF	VARIAN PART NO	DESCRIPTION
CR1	66-300457	Diode,Signal,1N457A
J4	51-407824	Conn,15 Pin,2 HV CAV
J504A	58-999724	Conn,MHV
J505A	58-028981	Conn,TNC
K1	72-569947	Relay,Dryreed,5V,1 Form C
P504B	952058-01	Cable,Assy,Dynode
P505B	963088	Cable,Assy,PbS
R1	32-201422	Resistor,Fxd,CC,2.2k,5%,1/4W
S1	71-711002	Switch,Rotary,3 Sec,2-5 Pos
	50-413207	Board,Cable Interchange,IRADS
	51-910251	HV Contact,Rt. Angle Plug
	23-508010	Knob

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Rear Cover
Pub. No. 50-413-206

USED ON: 2200/2300 Accessory
ASSY NO: 50-413-207